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## Final Report

### Multidisciplinary University Research Initiative on **REVOLUTIONARY MATERIALS FOR HYPERSONIC FLIGHT**

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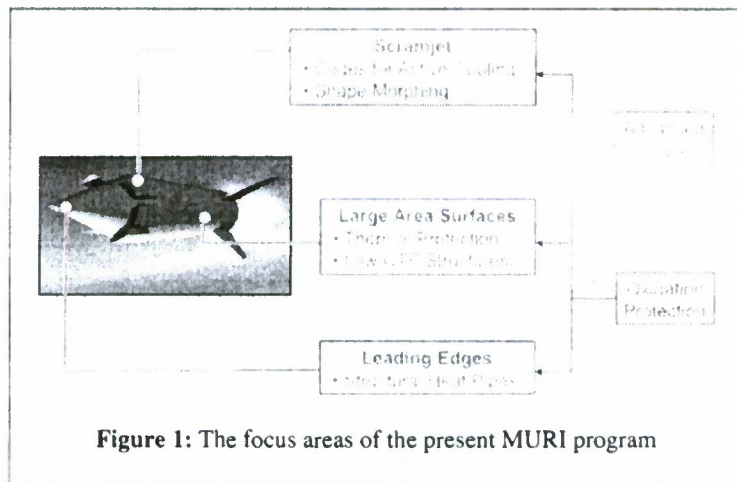
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**Abstract:** A team had been assembled to address challenges presented by future generations of hydrocarbon-fueled, long-range expendable missiles that operate up to Mach 7. The core issues involve robust materials and designs capable of sustaining pressure loads and extreme heat flux as well as shape-morphing components that allow efficient flight scenarios. The concepts invoke active cooling of the combustion system as well as passive cooling, through the use of heat pipes, of leading edges. The emphasis is on refractory alloys, coated to provide oxidation protection. Structural concepts leading to near-zero thermal expansion are also pursued. The document highlights the principal challenges and the key accomplishments of the program. Additional details are found in the published papers, listed at the end of the document.

## 1. THE RESEARCH OPPORTUNITIES

The extreme conditions present in hypersonic vehicles lead to unprecedented design challenges. The most critical regions include the scramjet engine, acreage surfaces, and leading edges (Figure 1).

(i) **Scramjet engines:** For air breathing vehicles, engine performance is strongly affected by shapes and contours of the compression ramp as well as the inner surfaces, including the inlet, the isolator, the combustor and the nozzle. Consequently, a contour that is designed for optimal operation at one Mach number may be inefficient at other Mach numbers. Surfaces that can *dynamically change shape* would greatly impact engine performance.



The success of air-breathing vehicles relies further on the development of systems that can survive the extreme thermal and mechanical loads associated with the propulsion system. *Active*



*cooling is essential* for survival. The design challenges are compounded by the multitude of constraints, including: temperature restrictions on the material and the fuel (coolant); material stresses arising from temperature gradients as well as pressures from the fuel and the combustion chamber; limitations on allowable component dimensions; and allowable pressure drops of the cooling fuel. They are further complicated by the non-linear coupling between optimal design and material properties.

(ii) *Acreage surfaces*. In areas of the vehicle that can only be passively-cooled, an overriding challenge is the management of the thermal strain differential between the hot outer surface and the cooler interior. Conventionally, this has been addressed by using oxide tiles that combine low thermal expansion coefficient with low thermal conductivity. The limitation is the inferior robustness of this system. An alternative approach envisages refractory alloys with topological configurations that yield a hot face with *near-zero thermal expansion coefficient* yet high stiffness and strength coupled with a core with low thermal conductivity but adequate shear and compressive strength.

(iii) *Leading edges*: The leading edges of hypersonic vehicles are subjected to intense, localized heat fluxes during atmospheric flight. The results are high local temperatures and severe thermal gradients. These conditions impose significant challenges to material selection. While metallic materials are favored for their durability to thermal distortion and structural deformation, their inherent thermal conductivities are too low to spread the heat effectively. One proposed solution is to use *heat pipes*. These rely on vapor flow for heat transport. The effective thermal conductivities that can be achieved are orders of magnitude greater than those of even the most highly-conducting engineering materials.

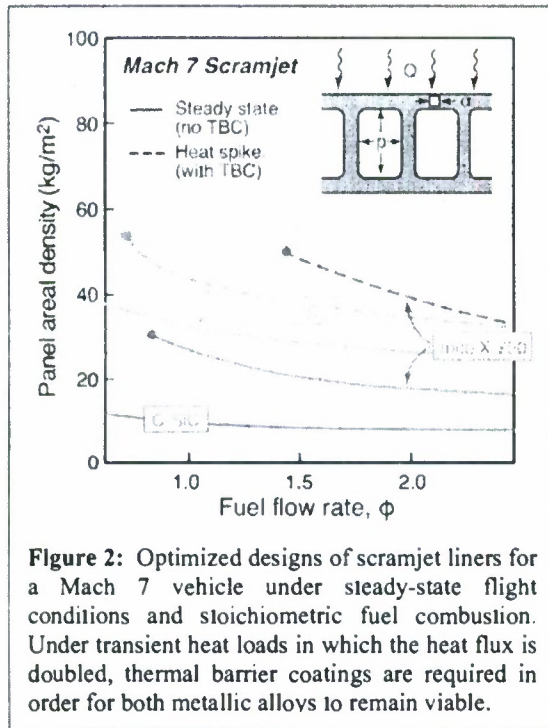
While metallic systems are desirable for use in hypersonic vehicles for durability and manufacturability, they often lack sufficient strength and oxidation resistance for the combustion environment. Furthermore, processing paths to manufacture the necessary structures are lacking, especially for materials with high strength. *New materials and processes as well as protective coatings for mitigating oxidation* are critical enabling technologies for hypersonic flight.

## **2. KEY ACCOMPLISHMENTS**

Materials and structures technologies with the potential to revolutionize hypersonic flight have been pursued within the present MURI. The emphasis has been on novel concepts for ameliorating the high heat fluxes and the resulting thermomechanical stresses, materials and processing routes to attain the desired component shapes with the requisite thermal and mechanical properties, systems that provide the flexibility needed for optimizing performance of the propulsion systems, and computational tools for analysis and design of actively-cooled components.

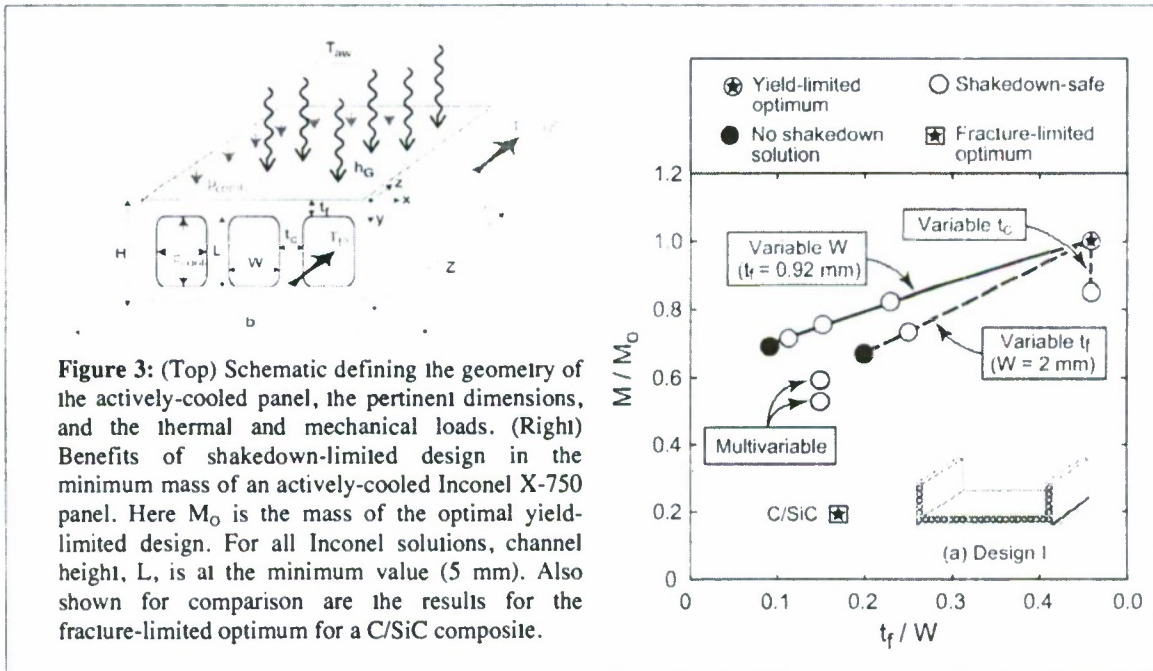
### **2.1 Design and Materials Selection for Actively-Cooled Scramjet Liners**

A materials selection methodology applicable to lightweight fuel-cooled combustor panels for scramjet engines has been devised and implemented [1,2]. The key ingredient is a code that can establish the capabilities and deficiencies of candidate designs and direct the development of



advanced materials. Additionally, the code allows direct comparison of the performance of candidate materials. The use of the code has been illustrated for a fuel-cooled combustor liner of a hypersonic vehicle, optimized for minimum weight subject to constraints on stress, temperatures, and pressure drop. Failure maps have been constructed for a number of candidate high-temperature metallic alloys and ceramic composites, allowing direct comparison of their thermostructural performance. Results for a Mach 7 vehicle under steady-state flight conditions and stoichiometric fuel combustion reveal that, while C-SiC satisfies the design requirements at minimum weight, the Nb alloy Cb752 and the Ni alloy Inconel X-750 are also viable candidates, albeit at about twice the weight (Figure 2). Under the most severe heat loads (arising from heat spikes in the combustor), only Cb752 remains viable. Property vectors that enhance design options have also been determined [3]. For one of the

promising candidate alloys (the Ni-based superalloy INCONEL X-750), the possibilities of reclaiming design space and lowering optimal combustor panel weight by tailoring its strength properties have also been assessed. The code has been provided to ATK, Boeing, AFRL, HRL, Teledyne, Northrop Grumman and Pratt&Whitney Rocketdyne.

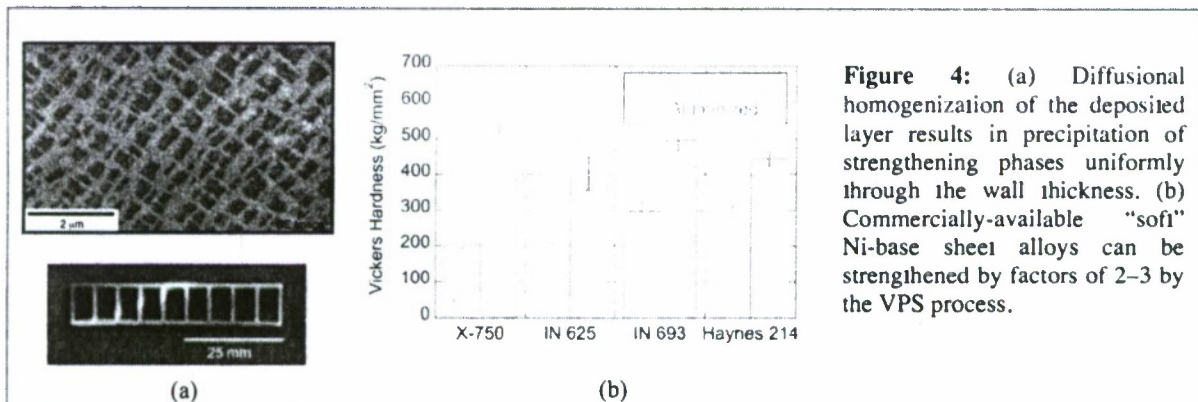




An approach that extends conventional yield-based design principles and utilizes concepts of localized plasticity and shakedown under cyclic loading in the design process has been identified and implemented [4,5]. For this purpose, a computational technique has been used to determine shakedown limits for prototypical cooled structures. An accompanying design sensitivity study demonstrates that, by allowing for shakedown, structures with areal densities significantly lower than those obtained from yield-limited design can be obtained (Figure 3). The magnitude of the benefits depends on the specific geometry of interest, the thermomechanical boundary conditions and the constraints placed on the design.

## 2.2 Vapor Phase Strengthening of Actively-Cooled Structures

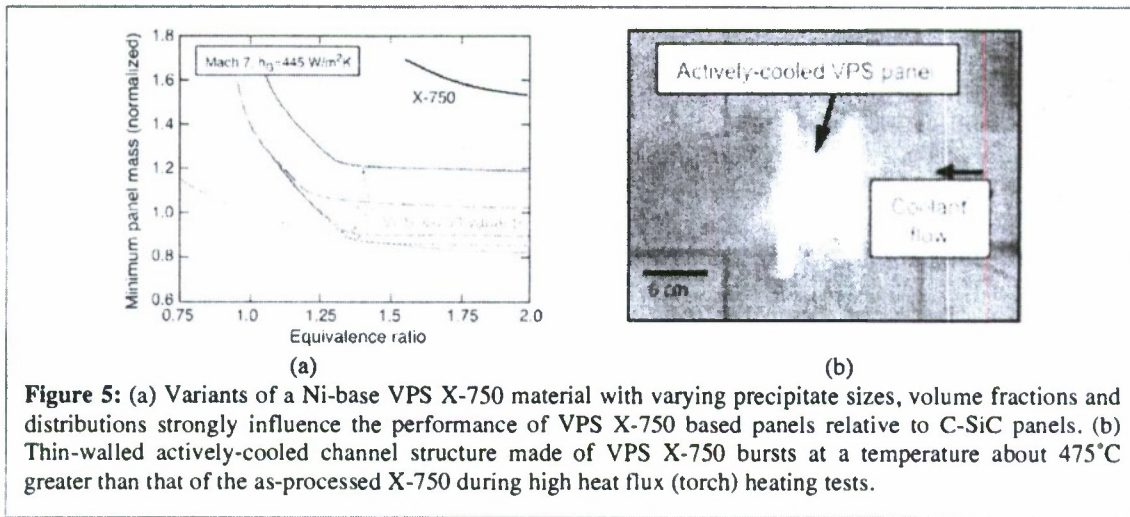
The preceding optimization code for actively-cooled structures has been used to define material attributes that would enable metallic materials to compete with C/SiC composites in scramjet engine applications. The results of that study motivated development of a new processing path for producing thin-walled structures from Ni-base alloys with significantly enhanced high-temperature strength.



**Figure 4:** (a) Diffusional homogenization of the deposited layer results in precipitation of strengthening phases uniformly through the wall thickness. (b) Commercially-available "soft" Ni-base sheet alloys can be strengthened by factors of 2-3 by the VPS process.

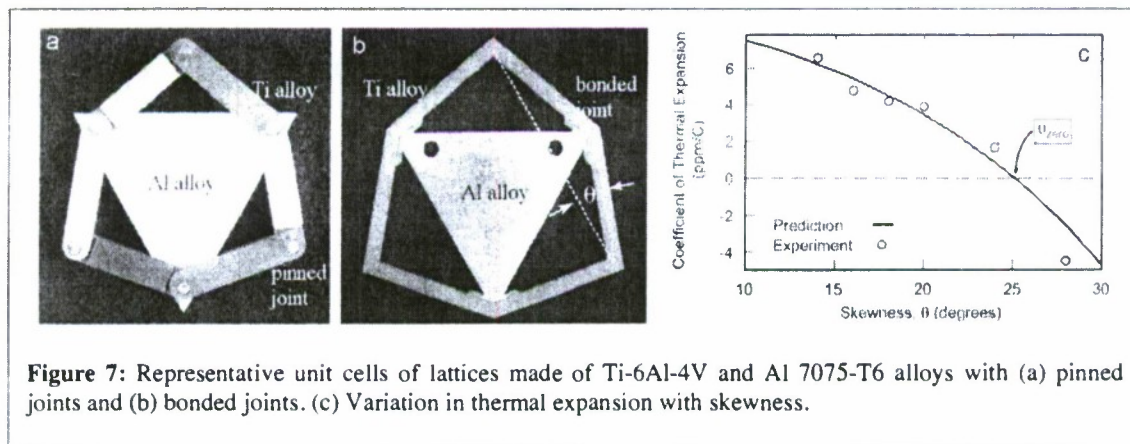
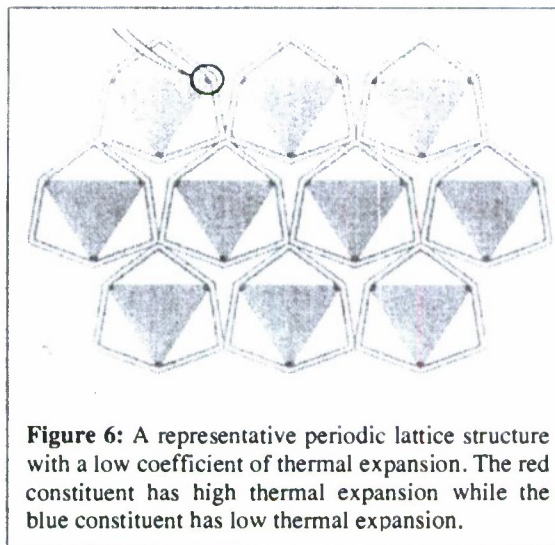
While metallic systems are desirable for durability and manufacturability, they often lack sufficient strength and oxidation resistance for the combustion environment. Furthermore, existing routes are not amenable to structures with exceedingly thin walls ( $<1\text{mm}$ ), especially for materials that are very strong at elevated temperatures. To this end, a new materials fabrication pathway for metallic alloys that are tailored to the unique property requirements for actively-cooled panels has been developed and demonstrated [6,7,8]. The approach consists of initial fabrication of requisite panel shape with formable, alloy-lean Ni or Nb sheet materials. Once shaped, the high-temperature strength and/or oxidation resistance are enhanced by incorporation of desirable elements, e.g. Al in Ni, using vapor phase processes, followed by high temperature homogenization. The diffusional homogenization of the deposited layer results in precipitation of strengthening phases uniformly through the wall thickness (Figure 4a). Models for strengthening mechanisms, alloy thermodynamics and processing conditions have been integrated with the new under high heat flux conditions (Figure 5b).

The new processing pathway provides unique materials solutions for structures with thin cross sections required for actively cooled structures. Models, details of processing paths and new materials design criteria have been provided to Boeing, P&W/Rocketdyne and AFRL.



### 2.3 Low Thermal Expansion Lattices for Thermal Protection Systems

A new planar lattice suitable for the hot face of a thermal protection system has been invented and demonstrated [9,10,11] (Figure 6). The design employs two materials with substantially differing thermal expansion coefficients, arranged spatially such that characteristic points on the panel are stationary during heating/cooling: whereupon the net thermal expansion of the panel is zero. The stationary points become locations where attachments can be affixed to a cold structure, thereby eliminating thermal stresses. Moreover, the lattice exhibits large stiffness to weight because it is fully triangulated and does not rely on rotational resistance at the joints for structural rigidity. A wide range of

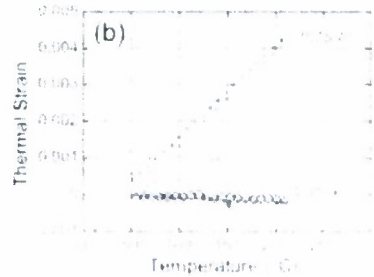
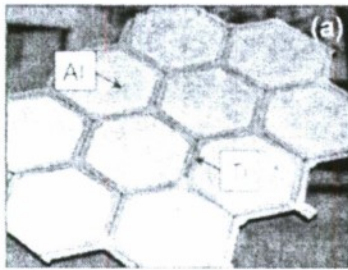




constituents can be used to build such lattices, enabling many desirable properties to be incorporated, especially high strength and toughness.

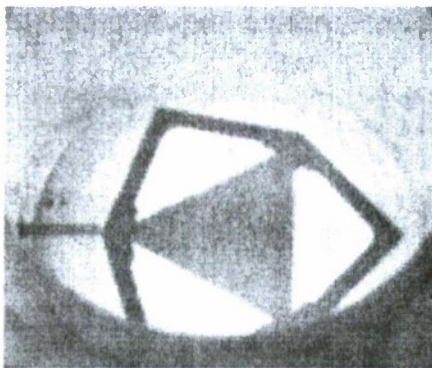
Unit cells of model systems of this type based on Ti and Al alloys are shown in Figure 7a and b. The two are distinguished by the nature of the joints:

either pinned or bonded. Experimental measurements and numerical computations (Figure 7c) confirm that zero thermal expansion can indeed be achieved through appropriate selection of the skewness angle (defined in Figure 7b). Modifications of the original lattice designs to enable larger skewness angles (and hence access a broader range of thermal expansion coefficients) along with higher stiffness and narrower gaps between the members have been implemented and analyzed (Figure 8).

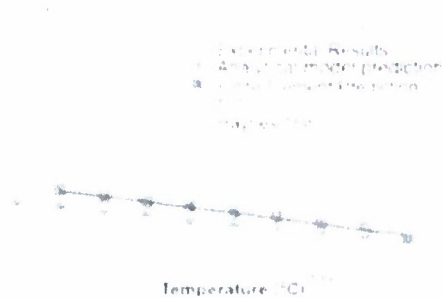


**Figure 8:** (a) An Al-Ti lattice with only extremely narrow gaps between members. (b) Experimental measurements and numerical computations confirm that near-zero thermal expansion is achieved.

Concepts for lattices with high temperature capabilities have also been devised [12]. In one case, the low CTE constituent consisted of a continuous network of the Nb-base alloy C-103 with inserts of high CTE Co-base alloy Haynes 188 (Figure 9). To ensure durability at elevated temperatures, a new coating approach developed within the program (described below) was employed. The coating comprises submicron alumina particles incorporated into Si-20Cr-20Fe. Thermal gravimetric analysis results indicate that the addition of submicron alumina particles reduces the oxidative mass gain by a factor of four during thermal cycling, thereby increasing lifetime. The result is a structure with a thermal expansion coefficient as low as  $1 \times 10^{-6} \text{ K}^{-1}$  at  $1000^\circ\text{C}$ : consistent with analytical models and finite element analysis predictions.



Coefficient of Thermal Expansion ( $10^{-6}/^\circ\text{C}$ )



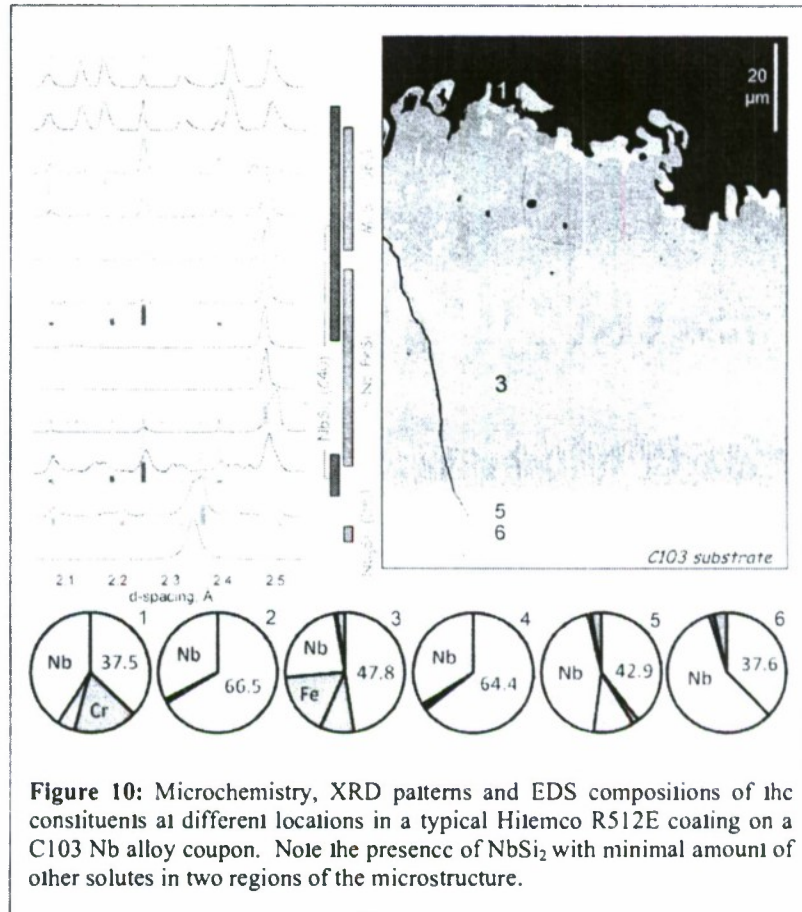
**Figure 9:** Low expansion lattice composed of silicide-coated Nb-base C103 alloy (outer element) and Co-base HA 188 (interior). The cell was repeatedly cycled to  $1000^\circ\text{C}$  and its expansion characteristics measured.

## 2.4 Oxidation Protection Coatings for Refractory Nb Alloys

Nb alloys have attractive thermal and mechanical properties but cannot be used without robust oxidation protection, currently enabled only by coatings. The industry standard is R512E (Si-13.4Cr-12.4Fe, at.%) which, upon combination with Nb, forms a complex array of silicide layers. Research within this program has focused on understanding the R512E coating in order to assess its suitability for hypersonic applications and to identify possible routes towards enhanced durability. The program undertook a rigorous microstructural

characterization exercise that led to a more accurate description of the system than previously available in the literature. Additionally,

an in-house processing capability for producing such coatings was developed, for the purpose of elucidating the mechanisms of microstructure evolution and for probing the efficacy of modified coating compositions in oxidation protection.



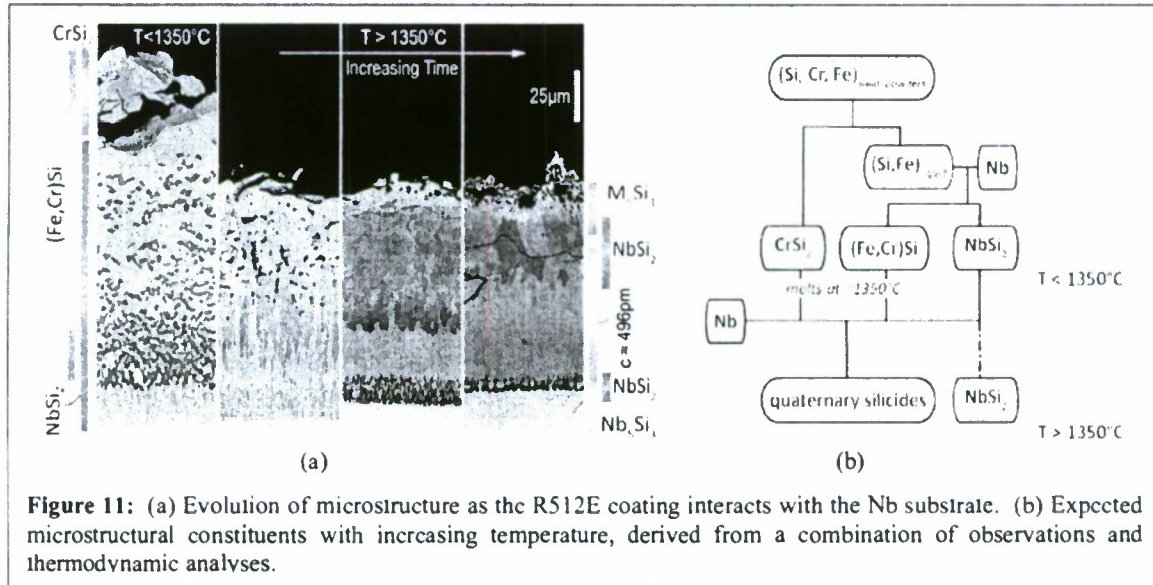
**Figure 10:** Microchemistry, XRD patterns and EDS compositions of the constituents at different locations in a typical Hitemco R512E coating on a C103 Nb alloy coupon. Note the presence of NbSi<sub>2</sub> with minimal amount of other solutes in two regions of the microstructure.

A major finding was the identification of the main constituent of the R512E coating (the lighter of the two thick layers in the middle of Figure 10): a highly textured quaternary silicide based on the structure of Nb<sub>78</sub>Fe<sub>40</sub>Si<sub>80</sub> (*P4<sub>2</sub>/mcm*), but with a composition closer to (Cr,Fe,Nb)<sub>6</sub>Si<sub>5</sub>, rather than the previously proposed Fe-rich M<sub>5</sub>Si<sub>3</sub> [13,14]. The other prominent layer is NbSi<sub>2</sub> with discontinuous M<sub>5</sub>Si<sub>3</sub> (*D8<sub>8</sub>*) at the surface. A thinner continuous layer of this phase is present below the quaternary *P4<sub>2</sub>/mcm* silicide, and two M<sub>5</sub>Si<sub>3</sub> layers of different structure and composition are also present at the top and bottom of the coating structure.

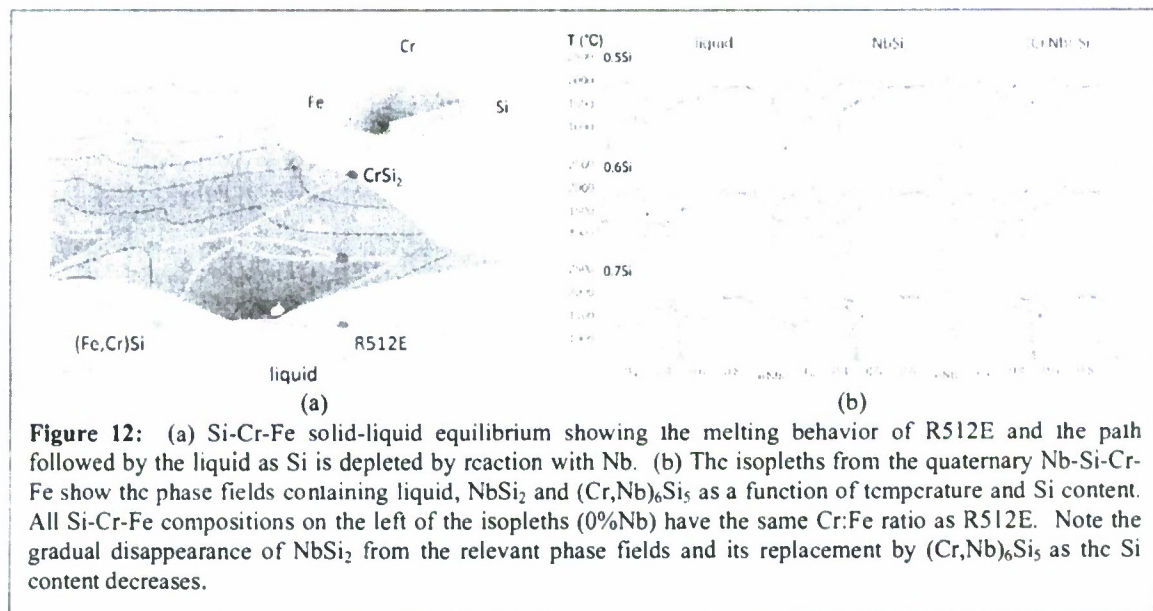
The development of in-house coating fabrication facilities and expertise, in conjunction with thermodynamic analyses of the Nb-Si-Cr-Fe system using the CompuTherm Pandat Nb database, was instrumental in elucidating the evolution of the multilayer silicide microstructure [15, 16]. The first event in its formation is the melting of the R512E (Si-Cr-Fe) mixture, leading to immediate crystallization of CrSi<sub>2</sub> and producing a Fe-Si rich liquid and elemental Si (Figure



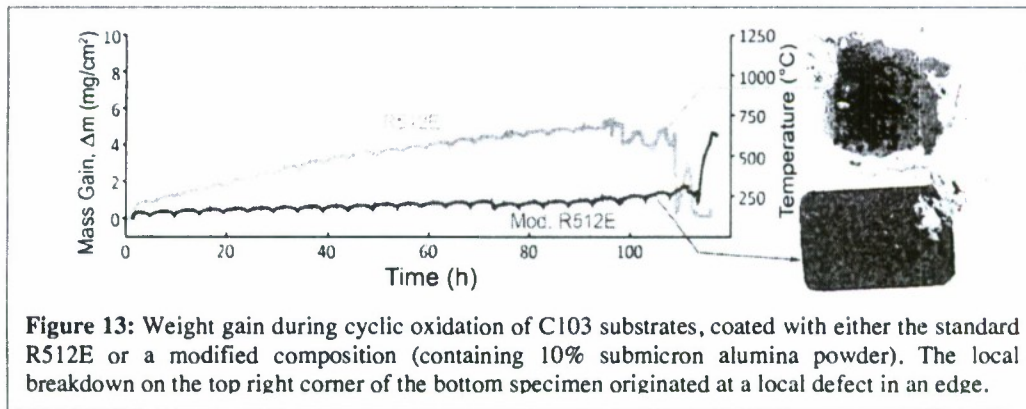
11). Thermodynamic calculations indicate the first liquid forms around 1163°C and, if Nb were not present, the Si-Cr-Fe mixture would be completely molten at ~1300°C (Figure 12). However, Nb from the substrate begins to dissolve into the melt upon contact, whereupon NbSi<sub>2</sub> precipitates, depleting the melt of Si and eventually inducing the crystallization of a mono-silicide (Fe,Cr)Si phase.



For heat treatment temperatures below ~1350°C, the coating is largely solid and consist of CrSi<sub>2</sub>, NbSi<sub>2</sub> and (Fe,Cr)Si (Figure 11a). This microstructure is distinctly different in constitution and morphology from that of the commercial coating (Figure 10). However, above 1350°C, the CrSi<sub>2</sub> and (Fe,Cr)Si phases interact to form a melt again, providing liquid for additional dissolution of Nb and the re-precipitation of higher temperature silicides. The quaternary isopleths in Figure 12b suggest that, as the Si content of the melt is depleted by the reaction with



Nb, the precipitation of a complex silicide of approximate composition  $(\text{Cr,Nb})_6\text{Si}_5$  becomes more favorable than the crystallization of  $\text{NbSi}_2$ . This phase grows highly textured, with the preferred orientation normal to the substrate. In this stage, time does play a more significant role, with the relative proportions of  $\text{NbSi}_2$  and the quaternary silicide depending on the hold temperature. Additional layers in the lower part of the microstructure form primarily by solid-state interdiffusion with the substrate. In essence, the critical event leading to the formation of the quaternary silicide  $(\text{Cr,Fe,Nb})_6\text{Si}_5$  isostructural with  $\text{Nb}_{78}\text{Fe}_{40}\text{Si}_{80}$  ( $P4_2/mcm$ ) takes place above  $\sim 1350^\circ\text{C}$ ; microstructures produced below this temperature are fundamentally different.



Isothermal and cyclic oxidation tests conducted on coated C103 and Nb substrates revealed that the R512E coating forms a slow-growing but non-passivating scale. This suggests that the improved oxidation resistance of the coated material relative to the bare substrate results from a reduction in the thermodynamic activity of Nb by its incorporation into the silicide, not from the oxide scale acting as a diffusion barrier. Cyclic oxidation leads to accelerated failures. In essence, the silicide phases have a higher CTE than the substrate so cracks open up upon cooling. These cracks oxidize on heating and the ensuing compression at high temperature drives them further into the coating. As they approach the substrate, they turn along the interface and link, leading to spallation. In searching for a possible approach to reduce the rate of crack propagation, it was found that addition of  $\sim 10$  vol.% sub-micron alumina to the R512E powder before processing leads to dramatically diminished weight gain and a significant delay in the onset of failure (Figure 13). Additionally, when the coating finally fails, the effects are less catastrophic than in the commercial material.

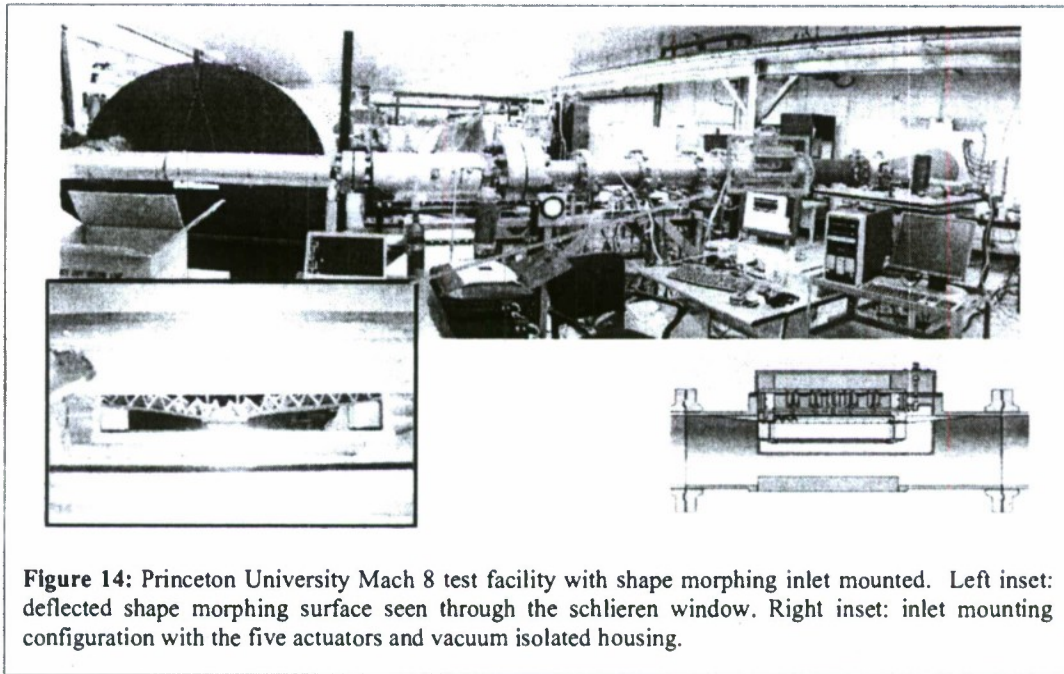
## 2.5 Shape Morphing Panels for the Hypersonic Flowpath

The development of advanced high speed flight vehicles is highly dependent on the ability to test performance. In the hypersonic regime, testing over a range of flight Mach numbers is needed in order to determine vehicle performance. This could be achieved through a shape morphing test facility, capable of dynamically changing Mach number during the test, thereby simulating portions of the flight regime. In addition, the shape morphing would allow the optimization of the test flow parameters for each flight configuration. A shape morphing concept has been demonstrated in the wind tunnel constructed at Princeton [17,18] (Figure 14). The system has been designed to maintain smooth flow through the tunnel as the Mach number is continuously



changed.

Materials for shape morphing structures in scramjet applications must be able to survive severe thermomechanical loads. They must be capable of withstanding temperatures in excess of 1500K for even modest Mach numbers (Mach 8) and be resistant to oxidation and fatigue. Additionally, in order for them to be effective in the hypersonic flowpath, their surfaces must be smooth. The structure should also support integrated cooling systems and provide effective thermal isolation from the structural components of the vehicle. For proper control it is important to have sensor systems embedded in the material so that feedback can be applied for optimum positioning.



**Figure 14:** Princeton University Mach 8 test facility with shape morphing inlet mounted. Left inset: deflected shape morphing surface seen through the schlieren window. Right inset: inlet mounting configuration with the five actuators and vacuum isolated housing.

These considerations led to the selection of a woven C-fiber SiC-matrix composite, recently developed at Teledyne. Use of a through-thickness angle interlock weave makes the material highly resistant to delamination. It is capable of operation to 1800K, can accommodate high thermal gradients (up to 1600K/mm), and is amenable to shaping into almost any geometric form. In addition, cooling ducts and holes can be integrally woven into the structure, facilitating insertion of sensors and use of transpiration cooling, without degrading strength.

For the present investigation, a 0.7mm thick C/SiC composite sheet was used to form the front surface of the structure, with actuators attached by integral pins woven into the composite [19]. The experiments were undertaken to determine the potential of this material for hypersonic morphing applications. Issues that were examined were the performance of the material under thermal stress, the impact of pressure gradients caused by start up and shut down transients, the stiffness and dynamic stability of the material in the presence of high frequency buffeting from hypersonic flow, and the ability of the material to deflect sufficiently far and sustain a specified contour sufficiently well to control the flow without leakage or fiber breakage. The experiments were conducted in a Mach 8 wind tunnel at a total temperature of 790K and at a stagnation

pressure of 1050 psi. Figure 14 shows the morphing surface installed in that facility along with insets showing the morphed surface itself and a diagram of the morphing inlet inserted into the top of the tunnel. The inlet was operated over a 6 to 1 area ratio change and achieved predictable control of the pressure contour along the centerline of the inlet with no material degradation. This result indicates that woven ceramic morphing structures can be implemented both in flight and for wind tunnel applications.

The successful tests have shown a viable approach to high temperature shape morphing. They have provided the basis for a new design of a shape-morphing hypersonic wind tunnel that will be capable of operating with continuous profile optimization in the Mach 4 to 6 regime. Follow-on work is underway to design such a facility.

## 2.6 Structural Heat Pipes for Leading Edges

Metallic heat-pipe leading-edge structures of the type shown in Figure 15 have been designed. The systems consist of a diverging vapor space with a structural I-core for load-bearing support. A methodology for their design was established through the use of models for prediction of thermal transport limits, heat capacity, and thermal performance under hypersonic heat fluxes. Thermal transport is dictated by three limits: (i) a sonic limit, encountered when the mean vapor

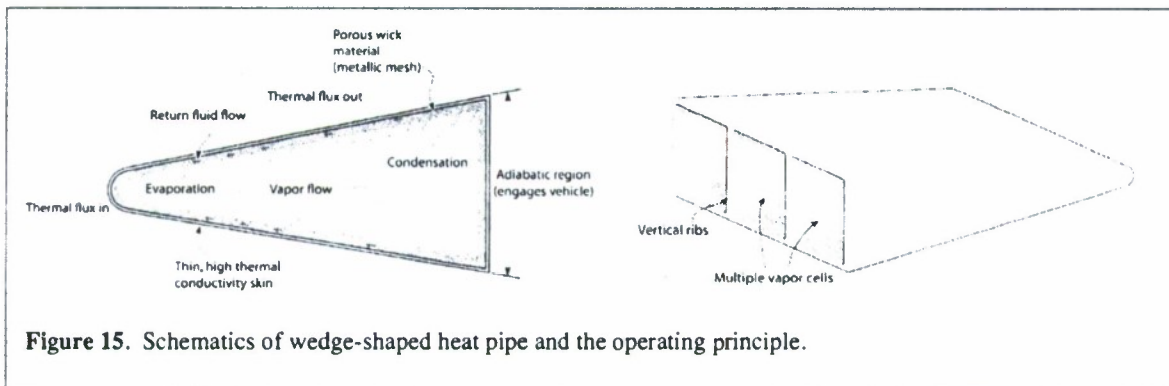


Figure 15. Schematics of wedge-shaped heat pipe and the operating principle.

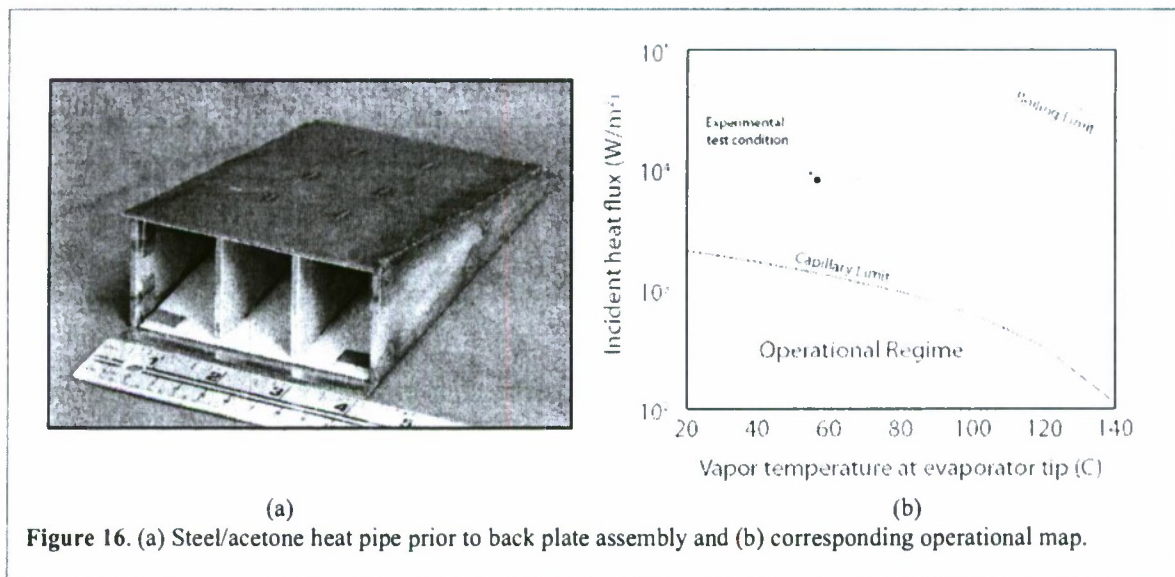
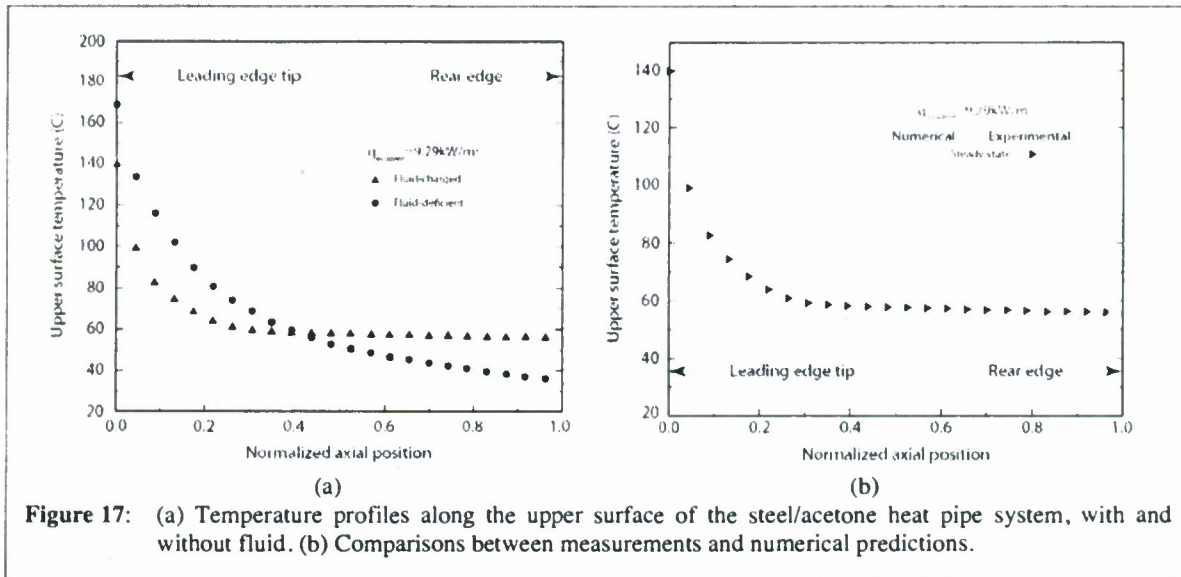


Figure 16. (a) Steel/acetone heat pipe prior to back plate assembly and (b) corresponding operational map.



flow velocity approaches transonic values; (ii) the boiling limit, which occurs when a critical superheating of the liquid is attained such that bubbles stabilize in the wick of the evaporator; and (iii) a capillary (pumping) limit, which arises when the drop in liquid and vapor pressure approach the maximum capillary pressure supportable in the wick.

A low temperature proof-of-concept heat pipe was conceived, fabricated and tested, for the purpose of validating the heat transport and thermal response models [20] (Figure 16). The case was fabricated from stainless steel and acetone was used as the working fluid. A stainless steel mesh was used for the wick. Comparisons between measured and computed temperature profiles for a high localized heat flux applied to the leading edge of the structure were satisfactory (Figure 17), although the tip temperature was slightly under-predicted: a consequence of partial dry-out due to the capillarity limit. The latter feature is evident on the operational map in Figure 16(b).



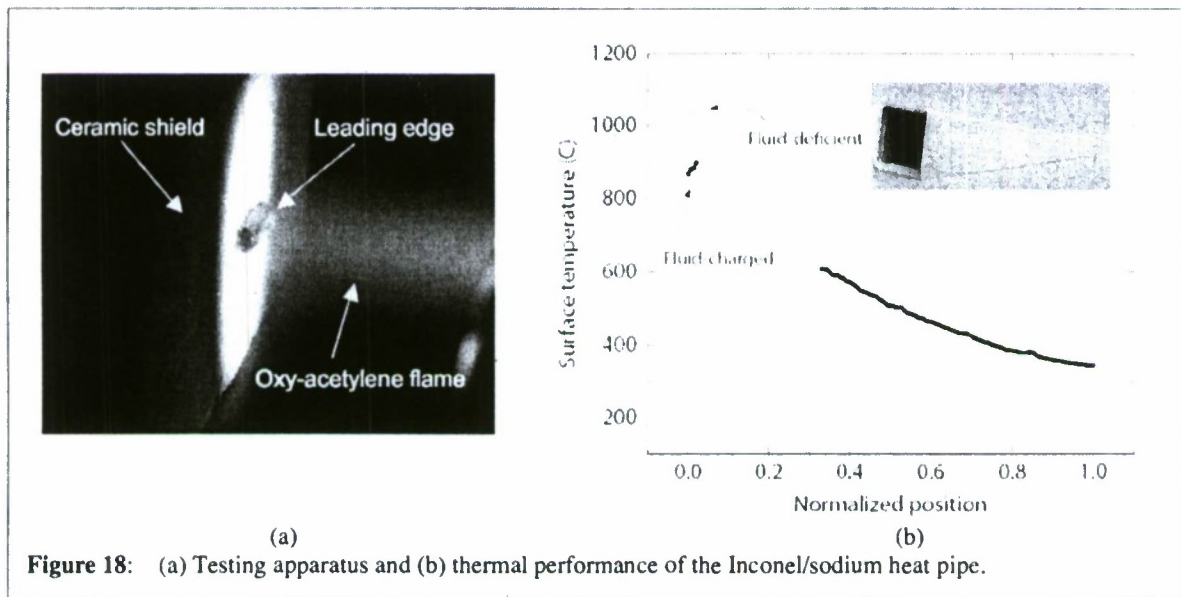
**Figure 17:** (a) Temperature profiles along the upper surface of the steel/acetone heat pipe system, with and without fluid. (b) Comparisons between measurements and numerical predictions.

Once validated at low temperatures, the models were used to design a high temperature system. The system consisted of an Inconel 718 case (with 3mm leading edge radius) and a nickel foam sintered to its upper and lower interior surfaces for the wick (Figure 18). Sodium was chosen as the working fluid. An oxy-acetylene torch was used to heat the tip and a porous alumina shield was used to deflect the heat away from the condenser. A thermal camera was positioned over the top face sheet to record surface temperatures. Temperature profiles show a reduction in peak tip temperature of about 130K (relative to an equivalent uncooled structure) and reduced temperature gradients.

Additionally, general design methods for a planar leading edge heat pipe have been developed, utilizing the leading edge thermal boundary conditions from standard hypersonic correlations [21]. The analytical predictions of the thermostructural performance have been verified by finite element calculations. Given the results of the analysis, possible heat pipe fluid systems have been assessed and their applicability to the relevant conditions determined. The results indicate that

the niobium alloy Cb-752, with lithium as the working fluid, is a feasible combination for Mach 6-8 flight with a 3 mm leading edge radius.

Effects of coatings on the exterior surfaces on the performance of structural heat pipes for leading edges have also been probed [22]. Two conclusions emerge: (i) Adding a thermal barrier coating does not allow operation at higher Mach number because the characteristic temperature of the system is unaffected by its presence. Instead, large (adverse) temperature gradients are induced through the coating. (ii) Coatings required to protect against oxidation can be safely applied provided that they are thin ( $<0.1$  mm) and have sufficiently high thermal conductivity ( $>5$  W/mK).





### 3. PUBLICATIONS

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1. L. Valdevit, N. Vermaak, F.W. Zok, A.G. Evans, 'A materials selection protocol for lightweight actively cooled panels', *Journal of Applied Mechanics* 75 (2008).
2. N. Vermaak, L. Valdevit and A.G. Evans, "Influence of Configuration on Materials Selection for Actively Cooled Combustors", *Journal of Propulsion and Power*, 26, 295-302 (2010).
3. N. Vermaak, L. Valdevit, and A.G. Evans, "Materials Property Profiles for Actively Cooled Panels: An Illustration for Scramjet Applications," *Metallurgical and Materials Transactions A; Physical Metallurgy and Materials Science* 40, 877-890 (2009)
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#### 4. STUDENTS

Name	Degree	University	Present employer
Bruce Alderman	M.S.E.	Princeton	
Jonathan Berger	Ph.D. (candidate)	UCSB	
Pamela Fetchko	M.S.	U. Michigan	Air Force Research Laboratory
Sara Johnson (Perez-Bergquist)	Ph.D.	U. Michigan	Los Alamos National Lab
Scott Kasen	Ph.D. (candidate)	U. Virginia	
Chris Limbaugh	Ph.D. (candidate)	Princeton	
Mark Novak	Ph.D.	UCSB	UCSB (post-doctoral fellow)
Nick Reese	M.Sc.	UCSB	Makai Ocean Engineering
Robert Rhein	M.S.	U. Michigan	Teach for America
Katherine Timpano	Ph.D.	Princeton	Orbital Sciences Corp
Greg Toland	M.Sc.	UCSB	ATK
Brian Tryon	Ph.D.	U. Michigan	Pratt and Whitney
Natasha Vermaak	Ph.D.	UCSB	UCSB (post-doctoral fellow)

#### 5. POST-DOCTORAL FELLOWS

Name	Present employment
Craig Steeves	Assistant Professor, University of Toronto
George Yu	
Chris Mercer	National Institute for Materials Science (NIMS), Japan
Ming He	Retired
Stephan Krämer	Senior Development Engineer, UCSB
Lorenzo Valdevit	Assistant Professor, University of California, Irvine

#### 6. HONORS AND AWARDS

A.G. Evans, Fellow of Imperial College (London)

A.G. Evans, C.G. Levi (D.R. Clarke) 2008 NIMS Award for Breakthroughs in Materials Research for Energy and the Environment, specifically for contributions to the *Enhancement of the Fuel Efficiency of Advanced Aero and Power Turbines through Materials Innovation* (Tsukuba, Japan).

R.M. McMeeking, Plenary Lecturer at the American Society of Mechanical Engineers International Congress, Boston, November 2008.

R.M. McMeeking, South West Mechanics Lecturer, February 2009.

T.M.Pollock, Fellow, The Minerals, Metals and Materials Society (TMS), 2009.